

**DETERMINATION OF POROSITY OF MATERIALS
BY MEANS OF METALLOGRAPHIC METHODS WITH
A HELP OF APPLICATION PROGRAMS**

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Abstract. Theoretical and experimental investigations of porous permeable materials of saponite-titanium and saponite-aluminium composites are presented in this work. Their qualitative and quantitative indexes are determined in PhotoM and Smart-eye applied programs. Metallographic images are presented by combination of structural components with different proportions: phases, shape and color, grain boundaries. The limits of technological parameters and synthesis of porous permeable materials on the basis of saponite-titanium and saponite-aluminium composites with the necessary balance of the designed and functional indexes and their variation with changes in individual properties have been studied.

On the basis of results of the investigations, regimes of shaping of saponite-titanium and saponite-aluminium composites are stated and substantiated. Dry radial-isostatic compaction and self-propagating high-temperature synthesis are applied. The porosity for these composites is determined. Recommendations on application of porous permeable materials are developed; and the branch of their application is determined: filtering porous permeable materials of saponite-titanium ensure the necessary purification and tertiary treatment of water for drink preserving their main natural characteristics: balance of chemical composition, organoleptic indexes, safety, physiological full value, and biological stability; and the filtering porous permeable materials of saponite-aluminium composite ensure treatment of sewage, of service and process waters. The suggested and tested resource-saving technology has been introduced in enterprises of the municipal services "Lutsk Vodokanal" and PAT "SKF-Ukraine" of Lutsk city; this technology provides manufacture of saponite-titanium composites for purification of water for drinking and saponite-aluminium composites for treatment of sewage and service and process waters; and developed, manufactured and tested porous permeable materials satisfy the requirements of state in the field of water supply, as well as in the food and industrial sectors, that, in its turn, plays an important role in solving complicated problems of modern materials science.

Introduction

With a help of different programs – PhotoM 1.21, Smart-eye, ScreenMeter 1.0, Micam, and others – it is possible to determine certain characteristics, which are necessary for qualitative and quantitative estimation of the structure of any material; and, in particular, porous permeable materials. Nowadays, high requirements are put concerning ensuring materials saving in different productions of machine parts, especially when expensive non-ferrous metals and alloys are used. Due to the given applied programs, their qualitative and quantitative characteristics are determined, and reduction of consumption of metal, energy saving, obtaining materials with special properties are ensured.

Problem statement

In to-day's stage of development of materials science, the manufacture of filtering materials by means of methods of powder metallurgy is one of competitive and energy-saving trends. To make filtering

elements cheaper, materials of saponite-titanium composite were substitute for titanium. To determine qualitative and quantitative characteristics of porous permeable materials, special applied programs – PhotoM 1.21, Smart-eye, ScreenMeter 1.0, Micam – are used. The application of these programs is considered to be expedient because they play an important role in solving complicated problems of modern materials science, enabling us to quickly estimate properties of material, to choose rational and specific conditions for their work that, as a result, leads to reduction in cost price of products, reduction in material consumption with, at the same time, increase in longevity.

Analysis of modern information sources on the subject of the article

The porous permeable materials are obtained by means of methods of powder metallurgy. For their sintering, traditional ways or self-spreading high-temperature synthesis are applied. The porous permeable materials which obtained by means of the self-spreading high-temperature synthesis, usually, have the following advantages: high mechanical strength, chemical and thermal stability, porosity, permeability, uniformity of pores, and other properties. To investigate these properties, the application of modern applied programs is necessary. The analysis of literature sources indicates that the application of applied programs calls for more profound theoretical and experimental investigation. Valuable contribution into development of this work are made by the following foreign scientists: R. German, W. Baek, J. Dillar [1], A. Sorokina, V. Savich, A. Galkin, V. Kaptsevich, L. Pilinevich [2] and others; also valuable contribution is made by the following Ukrainian scientists: Z. Duriagina [3], Yu. Zhyhuts, V. Lazar [4], V. Rud', V. Savuliak, L. Samchuk [5], and others.

Statement of purpose and problems of research

The aim: to determine qualitative and quantitative characteristics of porous permeable materials with a help of modern applied programs.

The tasks:

1. With a help of PhotoM 1.21 program, to carry out investigations of samples of saponite-titanium and saponite-aluminium composites sintered by means of the method of self-spreading high-temperature synthesis and by means of furnace method.
2. With a help of an optical eScope microscope, to verify the technique of determination of porosity of saponite-titanium and saponite-aluminium composites.
3. With a help of Smart-eye product, to carry out morphological analysis of the surface relief of specimens of porous permeable materials.

The main material presentation

Metallographic images can be presented by a combination of different structural components of different proportions: phases which are characterized by different sizes, by their forms and color, as well as by grain boundaries, which can be presented by either individual lines in an image or they can cover the image in the form of continuous network. A combination of these structural components may give us a very complicated picture; therefore, the main requirement to qualitative analysis of images can be stated and set in this way: in an obtained by means of a microscope photograph, the structural components are to be distinguished, then they should be classified with respect to their luminances, sizes, and shapes. Practical implementation of this problem contains the following (which have already become classic ones) subproblems: segmentation, filtration of faults, distinguishing the objects from background, patterns recognition. For successful conduction of metallographic analysis, the issue of reliability of segmentation of image remains to be the main. Because of complicacy of metallographic images, there is no possibility to determine characteristics of the object preliminarily. Therefore, the process of segmentation must be adaptive and, if possible, all the objects in an image which are of interest, no matter what their sizes or brightness are, are to be distinguished. With this, the possibility for the operator to interfere in the process of recognition, at least for correction of an object, must be remained.

The modern stage of software, is characterized, alongside with the increase in functionality, by the following tendencies:

- simplicity of operation;
- increasing the productivity by the system itself;
- reduction of requirements to the professional level of user.

Nowadays, there exist many different programs for analysis of images. The products which are the most understandable in operation become the most successful ones.

With taking into account their functional possibilities, among different software for image analysis, the most successful are the following programs: PhotoM 1.21, ScreenMeter 1.0, Micam. The program PhotoM 1.21 is created for cytophotometry. The program carries out the calculation of optical density of photographs. It is possible to load black-and-white images in BMP and JPG formats and to change the scope of viewing. Optical density can be calculated with taking into account the background both as to the average (delimited domain) and as to an individual photograph. Besides, it is possible to subtract dark field of video camera. Besides the calculation of optical density, inverting, increasing the contrast and smoothing the image, determination of the distances between objects and areas of regions in a photograph are provided. the regime of calibration for converting all the coordinates into metric units is also provided.

On the basis of the algorithm [6], these programs enable us to calculate average brightness of each object according to the scale of brightness which is determined in the system. With a help of the given scheme, in the aforesaid programs for analysis of images, the following sequence of algorithms for processing and obtaining characteristics of metallographic structure is suggested:

1. filtration of an image in order to exclude random noise.
2. Preliminary segmentation which is aimed at singling out the homogeneous regions.
3. Correction of an object in order to determine the threshold of brightness.
4. Final segmentation with a use of the determined background value.
5. Analysis of the singled out objects in order to determine their parameters.

Generally, qualitative analysis of images is conducted in order to determine the following parameters of an object: average brightness, perimeter, area, minimal and maximal diameters (of particles), factor of shape, coefficient of shape, and others. with a help of the aforesaid application programs, it is possible to determine characteristics which are necessary for qualitative and quantitative estimation of the structure of any material and, in particular, porous permeable materials. Nowadays, enhanced requirements are put to ensuring materials saving in manufacturing of machine parts, especially when expensive non-porous metals and alloys are used. One of the ways of ensuring this requirement is the manufacturing of machine parts according to the method of powder metallurgy. They ensure the reduction of metal losses up to 60 % for mechanical working, reduction of energy expenditures in manufacturing of machine parts from hard and heat-resistant alloys, obtaining of materials with special properties [7].

Properties of porous materials depend on properties of the initial powders and on the technological process of their manufacturing. The manufacturing scheme of the method self-propagating high-temperature synthesis created in Lusk National Technical University is given in Fig. 1.

The technological process is characterized by the following main parameters:

- limited number (in majority of cases – no more than three) of main operations with inconsiderable amount of preparatory and auxiliary operations;
- the most complete procession of initial materials into final product (waste-free) with small need in auxiliary materials;
- distinctly profound process stages of initial materials, in which drastic changes of their structures and properties in the course of main operations take place, often with change of aggregate state;
- maximal readiness of the final product article to operation or inconsiderable final working required (the process enables us to obtain such final product in the course of main operations);
- domination of energy consumptions for basic operation (operations) in in general balance of energy consumption.

For their manufacturing, metal powders both of spherical and non-spherical shapes of particles are used; the sizes of particles can be from several to one thousand micrometers, they can be of different

metals and alloys. The technology of manufacturing the porous metals from metal powders contains the traditional for powder metallurgy scheme of operations: shaping and sintering. The main requirements which are put to shaping in manufacturing the porous materials are related to obtaining articles of the given shapes and sizes with the necessary porosity and with ensured uniform or of given distribution of porosity and proper permeability. To increase the porosity and to facilitate the compacting of powders, special fillings are added to the metal powders; the fillings meet the following requirements: their decomposition does not take place at room temperature, no chemical interaction with the metal powder during mixing and compacting, their decomposition occurs at a temperature less than the temperature of sintering, no product of decomposition remains in the pores of the work piece after sintering. The amount of the filling, usually, is from 3 % to 10 % of the mass of the metal powder.

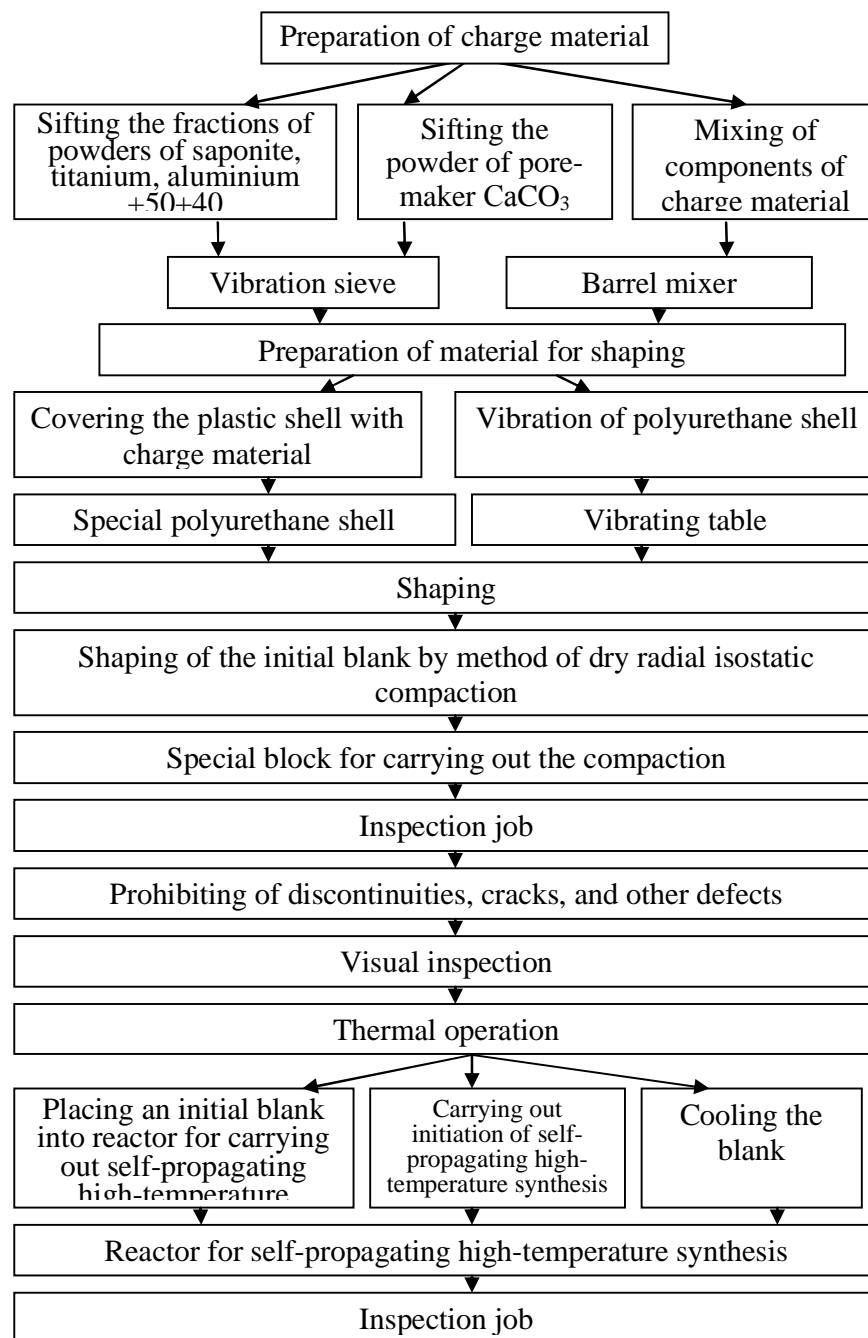


Fig. 1. Manufacturing scheme of self-propagating high-temperature synthesis created in Lusk National Technical University

Works in investigation of porous permeable materials are being activated by great potential possibilities of their application for production of filters, membranes with partial or full permeability. Titanium and aluminium are materials of prospect for production of porous structures because these materials are of high strength, resistant to corrosion, and refractory (high-melting). A complex investigations aimed at the study of possibilities of obtaining materials from these elements have been carried out. The limits of technological factors (composition, grading, regime of annealing) which are determinative in formation of structures of the desired porosity and the necessary mechanical properties, first of all strength. Synthesis of porous materials on the basis of titanium and aluminium with the necessary balance of the rated and functional indexes requires detailed investigation of the laws of formation of bonds in the composition-structure-properties system and their variation with changes in individual technological factors. For implementation of the stated tasks, it is necessary to carry out analysis of the influence of the conditions of their treatment; the analysis being concerned such characteristics of specimens as sizes of structural elements, variety of pores, and their distribution with respect to their sizes.

The initial specimens in the form of cylinders were shaped according to the method of radial-isostatic compaction at a pressure of 760 MPa with the initial density of 0.87-1.5 g/cm³.

The filling of CaCO₃ (Fig. 2) was chosen from the following reasons:

1. The optimal pore-making additives are those which in the course of sintering decompose without liquid phase at a temperature below that of sintering and without formation of composites which cause impurity of the main material.
2. The pore-maker must work as a lubricant and a binder reducing influence of the friction in compaction, and thus it must improve qualities of compacting and shaping.



Fig. 2. Calcium carbonate, CaCO₃ (ГОСТ 8253–79): general view (a), burnout (b)

In order to determine the optimal amount of pore-maker which is necessary for good formation of the system saponite-titanium and saponite-aluminium, we have carried out experimental investigations on the determination of the influence of the fraction of CaCO₃ on pore formation of these systems.

Taking into account the fact that the characteristic of compaction of powder is the ability of the latter to remain the shape which is given to it in the course of compacting and with which the specimen does not crumble and has no separation into layers after it is removed from the mold; as well as the fact that the greater its porosity is, the higher its permeability is, we have chosen the minimal value ($\sigma_{cr} = 1$ MPa) of compressive strength, and of the pressure of compacting was within the range 350–760 MPa. Analysis of the results of investigations indicates that the minimal content of pore-

maker considerably improves properties of compacting the investigated powders, and it amounts 5–6 %. Therefore, on the basis of the given results, in compacting the workpieces of porous permeable materials, no less than 4 % of CaCO_3 were introduced into the initial charge material. The regularity of structure, which depends on the properties of the initial powders and on the technology of manufacturing the porous permeable materials is an important characteristic of a porous material. Faultless results of investigation of any technological regimes of manufacturing concerning properties of porous permeable materials can be obtained only in the case when the porous structure is regular; and for this purpose, it is necessary to know the critical thickness of the porous permeable materials at which such regularity can be achieved. One of the parameters of regulation of properties of porous permeable materials which are obtained by compacting is the pressure of compacting. It is investigated that at a pressure above 300 MPa, the porosity does not change with a change in pressure, and it is equal for powders of different fractions. At a pressure below 300 MPa, the porosities of specimens, which are manufactured from larger particles, is greater. This is accounted for by the fact that the shape of large particles of powder is less spherical than that of smaller particles of powder. The greater the content of pore-maker is, the lower the strength of porous permeable materials is.

To achieve due strength, the specimens which are manufactured from a powder of larger fraction must contain no more than 25 % of pore-maker, and the specimens of less fraction more than 25 % [7]. This can be accounted for by different shapes of powder particles of small and large sizes, which essentially influences mechanical strength at a content of powder-maker less than 25 %. With a help of powder-maker, it is possible to obtain a previously set distribution of pores over a section of the specimen of a filtering porous material, this ensures good properties of performance. A pore-maker enables us to increase porosity of porous permeable materials and, respectively, their permeability and foul-capacity. Therefore, investigation of the influence of a pore-maker on performance characteristics of filtering porous permeable materials enables us to ensure its optimal amount for obtaining the desirable distribution of pores [8]. with a help of PhotoM 1.21 program, experimental results of specimens of saponite-titanium composites which are sintered according to the method of self-propagating high-temperature synthesis are shown in Fig. 3, *a*; those sintered according to furnace method in Fig. 3, *b*.

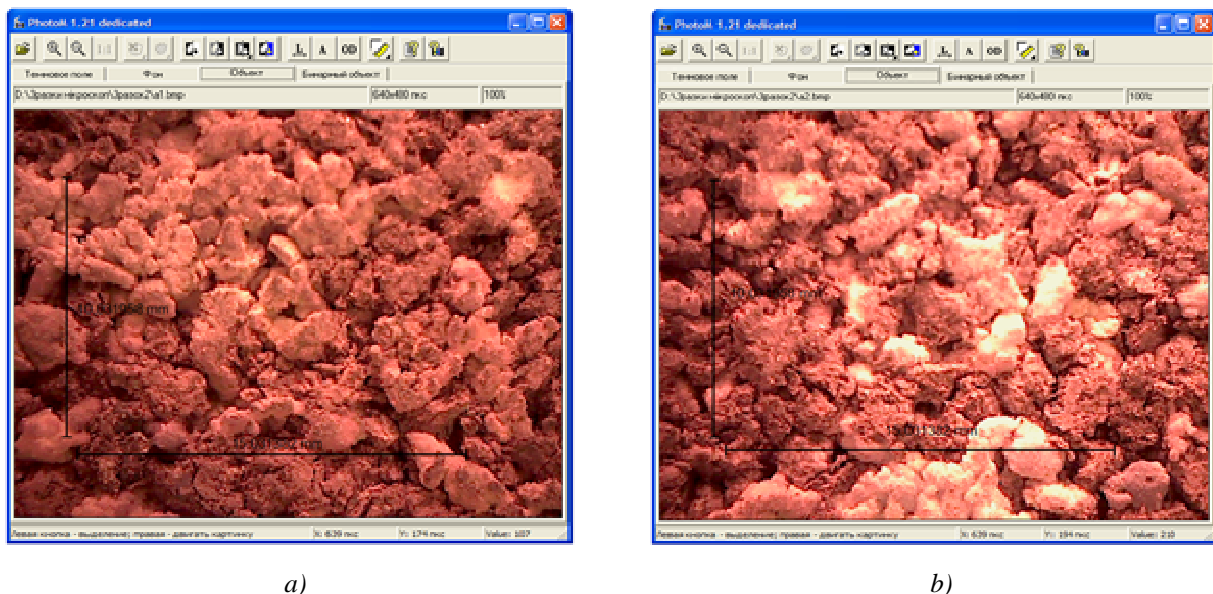


Fig. 3. Saponite-titanium specimens sintered according to self-propagating high-temperature method (*a*) and furnace method (*b*)

Results of porosity obtaining for a saponite-aluminium system are given in Fig. 4, *a*, for those sintered according to self-propagating high-temperature synthesis method, and in Fig. 4, *b*, for furnace method.

As an example and for checking the techniques of determining the porosity, we present the results of investigation of distribution of porous permeable materials of the composites which are manufactured from the powders of saponite-titanium (Fig. 5) and of saponite-aluminium (Fig. 6) with a help of an optical microscope eScope [9].

The results of investigation are given in Table 1. The truthfulness of the technique was estimated by means of comparison with the known results [10].

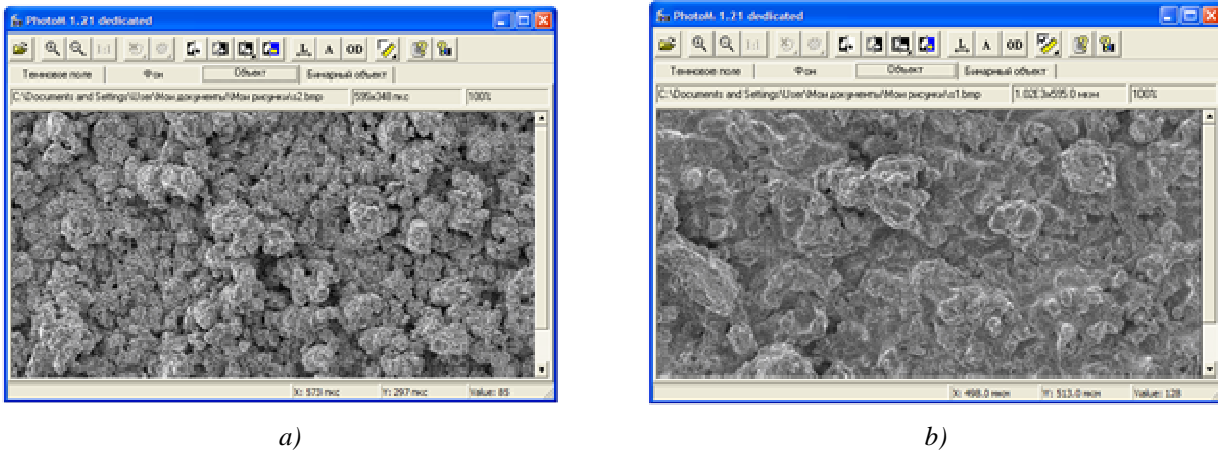


Fig. 4. Sintered specimens of saponite-aluminium, *x*1000. Self-propagating high-temperature method (*a*); furnace method (*b*)

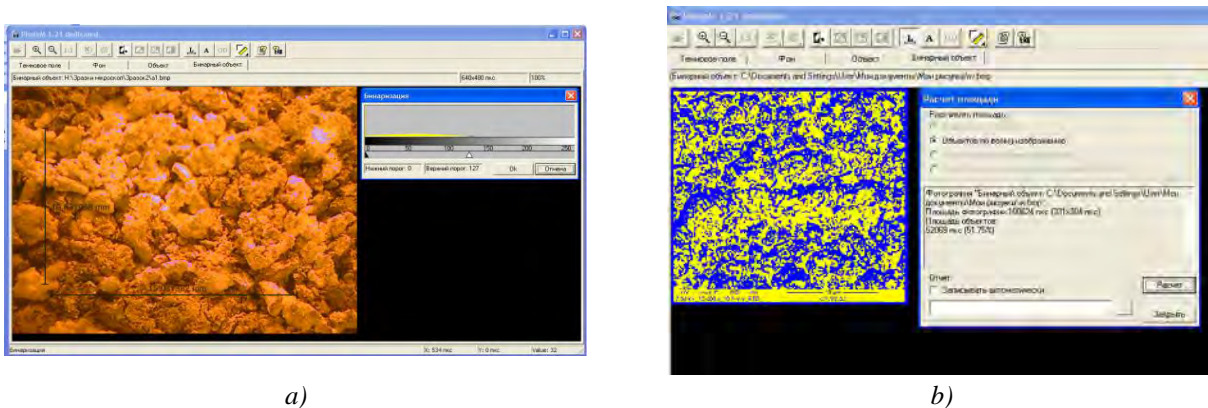


Fig. 5. Saponite-titanium “PhotoM 1.21” binarization (*a*), negative (*b*)

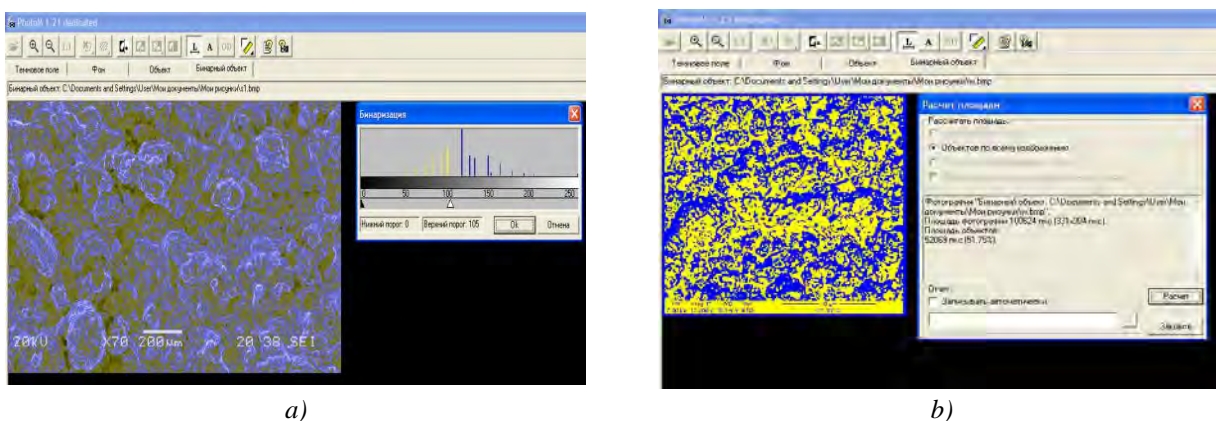


Fig. 6. Saponite-aluminium “PhotoM 1.21” binarization (*a*), negative (*b*)

Table 1

**Porosity of specimens manufactured from saponite-titanium
and saponite-aluminium composites**

Specimen	Technique of determination of porosity, μm					
	Method of whirl currents	Δ , %	Porous Materials, Inc	Δ , %	Metallographic method	Δ , %
Saponite-titanium	0,344	5,5	0,378	6,0	0,367	4,0
Saponite-aluminium	0,373	5,0	0,415	7,0	0,399	3,8

Δ is the error of measurement (compared to GOST 25281–82).

Conclusions

The applied programs PhotoM and Smart-eye enable us to ensure qualitative and quantitative indexes of porous permeable materials. On the basis of results of the investigations, the regimes of formation of saponite-titanium and saponite-aluminium components are stated and substantiated. For compaction of specimens 220 mm high and 40 mm in diameter, dry radial-isostatic compaction in the pressure range of 500–700 MPa was applied. The total time of sintering in the regime of self-propagating high-temperature sintering process for saponite-titanium is 80 s at the temperature 1350 °C and for saponite-aluminium 75.5 s at the temperature 1300 °C. The process rate of burning of obtained material is within the range of 0.46–3.67 mm/s and in the pressure range of 0.5–4.0 MPa. The porosity of saponite-titanium composite is within 35–40 %, and of saponite-aluminium composite 40–45 %. The filtering porous permeable materials of saponite-titanium composite ensure the necessary purification and tertiary treatment of water for drinking preserving their main natural characteristics: balance of chemical composition, organoleptic indexes, safety, physiological full value, and biological stability; and the filtering porous permeable materials of saponite-aluminium composite ensure treatment of sewage, of service and process water; that, in its turn, plays an important role in solving complicated problems of modern materials science. The suggested and tested resource-saving technology has been introduced in enterprises of the municipal services “Lutsk Vodokanal” and PAT “SKF-Ukraine” in Lutsk city; this technology provides manufacture of saponite-titanium composites for purification of water for drinking and saponite-aluminium composites for treatment of sewage and service and process waters.

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